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C.S. SATHYA MUKESH\*\*\*#**FABRICATION OF ALUMINUM – Cr<sub>3</sub>C<sub>2</sub> SURFACE COMPOSITES THROUGH FRICTION STIR PROCESSING  
AND ANALYZING ITS MICROSTRUCTURAL AND MECHANICAL EVOLUTION**

In this paper, aluminium alloy of grade ADC-12 was considered as a base metal and chromium carbide (Cr<sub>3</sub>C<sub>2</sub>) particles were reinforced through friction stir process. A detailed analysis of mechanical property and metallurgical characterization studies were performed to evaluate the surface composite. Remarkable changes were observed in the developed composite due to the mechanical force produced by the stir tool with an increase in hardness. The metallurgical investigation infers that the presence of silica in ADC-12 alloys has undergone mechanical fracture and long needle structure changed to reduced size. On the other hand, at higher tool rotational speed, the uniform distribution of hard particles was confirmed through SEM micrographs. Thus the modified surface composite has produced good mechanical property with high metallurgical qualities.

*Keywords:* microstructure, microhardness, Friction stir processing, ADC-12, Chromium Carbide

**1. Introduction**

Weight reduction is one of the major demands in the field of aerospace and automobile applications which urged researchers to prefer low weight materials like aluminum. The replacement of conventional metals like steel with aluminum alloy in these applications is highly anticipated. But the insufficient stiffness and strength offered by these alloys limited its applications. In order to overcome these drawbacks, it was necessary to reinforce the aluminium alloy. This led to the development of Aluminum Metal Matrix Composites (AMMC) which exhibits hardness and strength owing to the dispersion of hard reinforcing particles. Also, the high specific modulus, wear resistance, strength to weight ratio and fatigue strength attracted these materials in the fields of the automobile and aerospace applications [1-4]. In most of the researches, AMMCs are developed through stir casting, compocasting and squeeze casting where ceramics particles of oxides, carbides, borides and nitrides are effectively reinforced into matrix material [5]. But in these processes, the formation of interfacial reactions that takes place between the matrix material and reinforcement is unavoidable [6]. These interfacial reactions may sometimes lead to the reduction in certain bulk properties like ductility and toughness. So as to prevent the formation of interfacial reactions, the reinforcement is dispersed onto the surface of a material for certain depth such that the hardness and wear resistance of the surface can be improved without affecting the bulk properties of the metal matrix.

Surface composites can be developed through dispersing certain effective ceramics onto the surface of a matrix material by some enhanced surface modification techniques [7,8] like plasma spray method, electron beam method, laser melting etc. But the high heat produced during these processing techniques resulted in the formation of certain detrimental and intermetallic phases which may reduce the characteristics of the developed surface composites [9,10]. This created a big gap in the field of surface modification techniques which paved way for the development of a semisolid processing technique. Friction Stir Processing (FSP), which is inspired from friction stir welding methodology, is a semi-solid technique which works below the melting point of the matrix material and thereby reducing the chances for the formation of detrimental phases. FSP tool causes severe plastic deformation to the matrix material due to its stirring action, leading to the microstructural modification of the base metal along with the homogenous dispersion of the ceramic particles [11,12].

Chromium Carbide is mainly available in three crystal structures namely cubic, orthorhombic and hexagonal structure. These phases provide an enhanced oxidation resistance and strength and henceforth used in many applications where wear and corrosion resistance are obligatory [13]. The properties of chromium carbide coatings such as hardness, wear resistance, corrosion resistance and temperature stability make them suitable for coating metals in various applications [14-16]. Coating Cr<sub>3</sub>C<sub>2</sub> over the surface of metals has been often tried by

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researchers which makes this particle as a highly researchable material. But the dispersion of these particles as reinforcements was found to be rare and the surface dispersion of the same was observed nil which created the research gap in this study. However, dispersion of chromium carbide onto the surface of aluminum alloy was not analyzed to a great extent by researchers and in this study, a detailed research on the effect of FSP and volume fraction (vol. %) of chromium carbide reinforcements on aluminum alloy ADC 12 is carried out. Microstructural and mechanical characterization of the developed surface composites, dispersed with varying vol. % of  $\text{Cr}_3\text{C}_2$  is studied and analyzed in this research.

## 2. Materials and methods

Aluminum alloy of grade ADC 12, the matrix material in this study was procured and was sliced to the size of 100 mm  $\times$  50 mm  $\times$  6 mm through Wire cut EDM process. The chemical element analysis of ADC 12 performed by optical emission spectroscopy analysis, expressed silicon (10.2 wt. %) as the major alloying element along with the elements like iron (0.67 wt. %), copper (2.9 wt. %), manganese (.35 wt. %), magnesium (.21 wt. %), nickel (0.38 wt. %), zinc (0.79 wt. %), tin (0.11 wt. %) and the balance available is aluminum. The  $\text{Cr}_3\text{C}_2$  powder particles obtained were  $\sim 5.8 \mu\text{m}$  in size and were in the spherical or round form. SEM micrograph of the received  $\text{Cr}_3\text{C}_2$  is portrayed in figure 1 which was used in this research without any further processing. Average particle size was analysed using ImageJ software. Similar findings have been reported by selvakumar et al. [17].

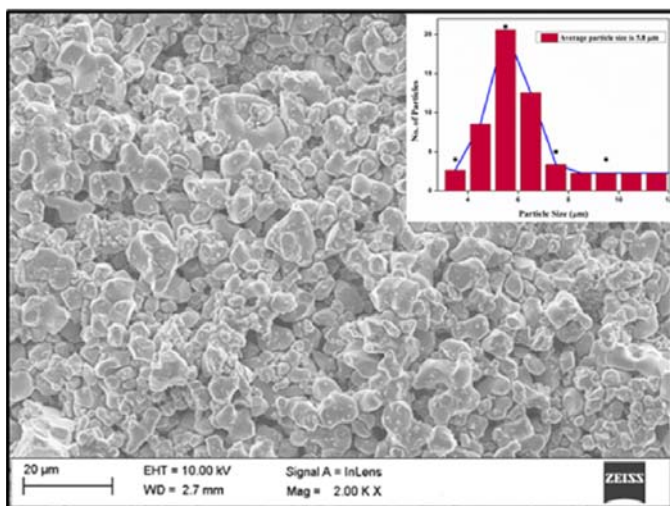


Fig. 1. FESEM micrograph of as received  $\text{Cr}_3\text{C}_2$  particles – inset histogram plot for particle size

Friction stir processing comprises of mainly three steps (i) cutting grooves on the centre of the matrix metal to compact the reinforcements, (ii) processing over the groove with a pinless tool to compact the powder and cover the groove so as to avoid

the scattering of particles during FSP route and (iii) FSP of the base metal to disperse the ceramics onto the surface of the matrix material. The processing process is provided in figure 2.

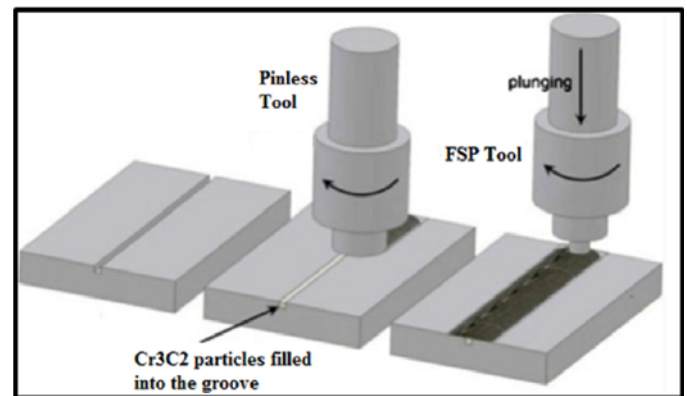


Fig. 2. Steps in friction stir processing [17]

In this research,  $\text{Cr}_3\text{C}_2$  are charged into the groove cut on the surface of ADC 12 aluminum alloys. A square groove was cut lengthwise over the aluminum plate for charging the particles at a depth of 2.5 mm with varied width based on the percentage of  $\text{Cr}_3\text{C}_2$  to be charged. The width of the grooves cut at the center of the plate were 0.35 mm, 0.7 mm, 1.05 mm and 1.4 mm respectively for 6, 12, 18 and 24 vol. % of  $\text{Cr}_3\text{C}_2$ . Into the groove, ceramic particles were charged and compacted. A pinless tool made of H13 steel was then allowed to pass over the groove so as to prevent the scattering of the particles from the groove during the FSP. During this process, the groove will be closed by the pinless tool. During the next stage of FSP, a FSP tool made of H13 tool steel with shoulder diameter 25 mm, pin diameter 5 mm and pin length of 3 mm was effectively used to modify the surface of ADC 12 aluminum alloy and thus disperse the ceramics over the surface of the base metal. FSP was done using a CNC milling machine whose displacement can be controlled. The process was carried out an optimized traverse speed and rotational speed of 30 mm/min and 1000 rpm respectively.

A microstructural analysis is an important step in the development of a composite as microstructure plays a major role in the bulk property of a material. Microstructural characterization of a composite material well defines the dispersion of reinforcement in the matrix material along with its bonding performance. Optical Microscopy (OM) of the developed Aluminum Surface Composite (AMC) along the direction perpendicular to the tool feed direction was carried out. So as to analyze the same, specimens were cut with the aid of WEDM and polished as per metallographic standards using grit papers of different grade and velvet polisher. Polished specimens were then etched using Keller's reagent, and the microstructure was analyzed using Field Emission Scanning Electron Microscope (FESEM) so as to analyze the bonding between reinforcement and matrix material. Energy Dispersive X-ray Spectroscopy (EDS) analysis of the developed surface composites has to be carried out so as to confirm the dispersion of reinforcement into the matrix metal.

Microhardness survey of the AMC dispersed with varying proportions of  $\text{Cr}_3\text{C}_2$  was analyzed with the help of a Vickers microhardness tester. Testing was carried out on the cross section of the specimen and also along the direction perpendicular to the tool traverse motion applying a load of 500 g for 15 seconds.

### 3. Results and discussion

#### 3.1. Microstructural characterization

The optical micrographs of the developed AMC reinforced with varying vol. % of  $\text{Cr}_3\text{C}_2$  are shown in figures 3(a-d), wherein a reduction in grain size can be observed. This fine equiaxed grain formation for the developed surface composites can be attributed to the dynamic recrystallization of metal that takes place as a result of FSP. During FSP of a metal, high temperature is produced due to friction created between the metal and tool shoulder which melts the metal and the tool pin stirs the pin breaking away the metal grains thereby reducing the grain size. The inoculations in Al alloys aids in the production of fine grains and thereby improves the mechanical properties. The presence of Cu, Fe and Mg forms intermetallic compounds. Increasing the cooling rates helps to refine the grain structure. The plate like morphology remains the same even the cooling rate refines Si particles. The phosphorus added to liquid alloy has an effect on the primary Si phase. When phosphorus is as low as 0.03% it aids in the refinement of grains. Figures also reveal a clear view of  $\text{Cr}_3\text{C}_2$  dispersed onto the surface of aluminum matrix.

Figure 3a and 3b infers the metallurgical changes of ADC-12 alloy before and after the FSP process. The structure of Si in the ADC-12 alloy was found to be protracted in unprocessed alloy. However, in the processed surface, the needle like Si has undergone fracture due to the rotation of the FSP tool. Further, it has been dispersed uniformly to the size of hard particle reinforced. The composite reinforced with finer particles show more % reduction in dendritic arm. Aspect ratios of dendritic arm for parent metal, volume percentage 6, 12, 18, 24

are 3.52, 3.02, 2.12, 1.30 and the percentage reduction is 14.2, 29.8, 38.6, and 19.25 respectively. Thus it can be concluded that the reinforcement plays an important role in reducing the aspect ratio of dendrites. Dendritic arm length (Si) decreases due to the rotation of FSP tool. Similar type of findings have been reported by Satish et. al, [18,19] and Nampoothiri et. al [20]. Reduction in grain size of FSPed surface of the base aluminum matrix can also be attributed to the presence of the  $\text{Cr}_3\text{C}_2$  in the matrix. The reinforced  $\text{Cr}_3\text{C}_2$  interrupts the growth of grains during solidification process thereby refining the same. It is evident from figure 4 that, with increase in the vol. % of reinforcement, the grain size of the matrix metal tends to reduce further. It can also be stated that the presence of  $\text{Cr}_3\text{C}_2$  in aluminum matrix disrupts the grain boundary movement thereby reducing the grain growth of the matrix material and this phenomenon can be otherwise called as the pinning effect which is also responsible for grain size reduction in the developed AMC.

SEM morphology of the developed AMC reinforced with varying vol. % of  $\text{Cr}_3\text{C}_2$  are portrayed in figure 5(a-d). It is evident from figures that the dispersed  $\text{Cr}_3\text{C}_2$  are distributed fairly in a homogeneous manner. This homogenous dispersion of reinforcement into the matrix materials might enhance the properties of the composite material. During FSP, the tool plastically deforms the aluminum base metal and due to the stirring action of the tool, this metal in the plasticized conditions is pushed back of the tool. And the same is forged into the groove created. The above said phenomenon is responsible for the homogeneous dispersion of particles on the surface of the thus developed AMC. The perfect distribution and bonding of the  $\text{Cr}_3\text{C}_2$  powder particles with the aluminium alloy was observed after microscopic examination of the FSPed samples. And little distortion in the particles was exhibited after FSP due to sharp edges of the Powder particles.

It can be noted from the SEM micrographs that the particle distance gets reduced with respect to the increased vol. % of  $\text{Cr}_3\text{C}_2$  in the aluminum matrix. This indicates an increment in the quantity of ceramics in the matrix. This can enhance the surface characteristics of the thus developed AMC. And the

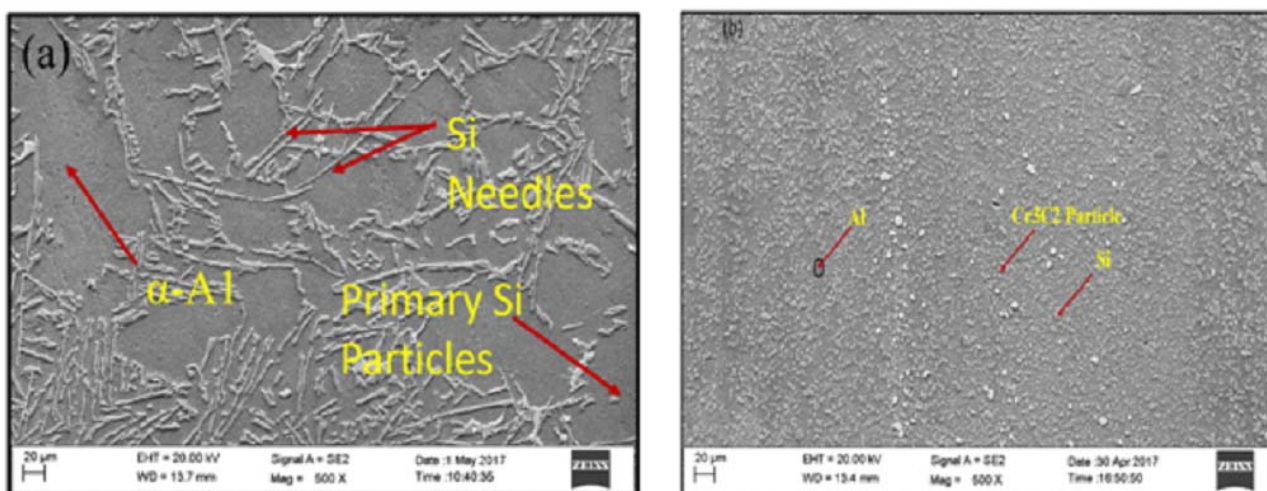


Fig. 3 (a) Parent Metal ADC-12 (Before FSP) (b) ADC-12 Reinforced  $\text{Cr}_3\text{C}_2$  after FSP

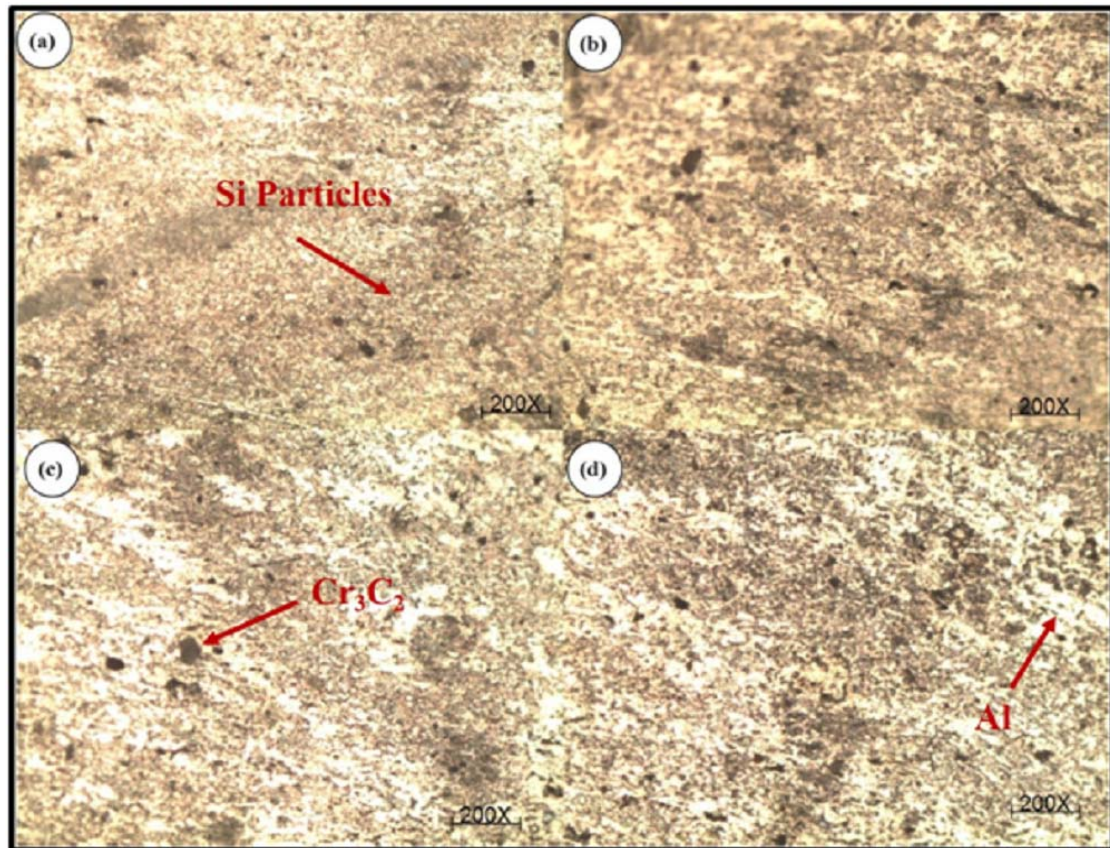


Fig. 4. Optical micrographs of FSPed aluminium surface composite reinforced with (a) 6 vol. % of Cr<sub>3</sub>C<sub>2</sub> (b) 12 vol. % of Cr<sub>3</sub>C<sub>2</sub> (c) 18 vol. % of Cr<sub>3</sub>C<sub>2</sub> (d) 24 vol. % of Cr<sub>3</sub>C<sub>2</sub>

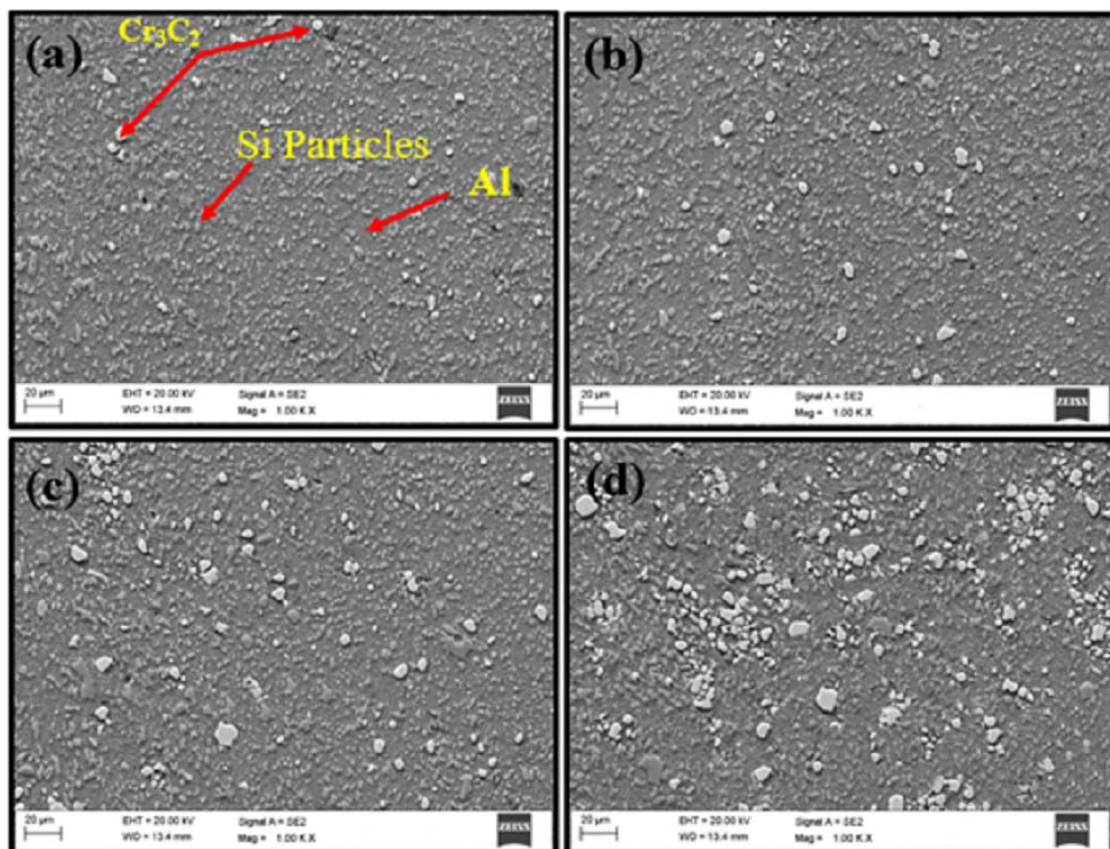


Fig. 5. FESEM micrographs of FSPed aluminium surface composite reinforced with (a) 6 vol.% of Cr<sub>3</sub>C<sub>2</sub> (b) 12 vol.% of Cr<sub>3</sub>C<sub>2</sub> (c) 18 vol.% of Cr<sub>3</sub>C<sub>2</sub> (d) 24 vol.% of Cr<sub>3</sub>C<sub>2</sub>

breaking down of particle size can be observed from the SEM micrographs. This non-uniformity in the size of  $\text{Cr}_3\text{C}_2$  dispersed onto the aluminum surface can be attributed for the high load exerted during the FSP route along with the vigorous rotating action of the tool leading to the breaking down of ceramics. A deep investigation on SEM results reveals that the ceramic particles exhibited good bonding with the aluminum matrix material. This can be proved with the fact that the presence of voids and cavities were absent in the SEM morphology and the formation of detrimental particles was also reported zero. The particle, aluminum matrix interface exposed nil cavities which also indicate the enhanced bonding created between the reinforcement and the matrix material.

EDS analysis of the developed surface composites was carried out and the results of AMC reinforced with 24 vol. % of  $\text{Cr}_3\text{C}_2$  is portrayed in figure 6b. The ADC12 aluminium alloy contains Si as the major alloying element and the microstructure examination shown in figure 3a ensures the presence of eutectic Si in needle shape and very few primary Si elements in the  $\alpha$  Al matrix. In addition, the presence of Si and various other alloying elements was also confirmed by EDS spectrum analysis and it

is shown in figure 6a. It is clear from the figure that, at the point of analysis the presence of  $\text{Cr}_3\text{C}_2$  was confirmed. It also gives a clear knowledge about the homogenous distribution of  $\text{Cr}_3\text{C}_2$  onto the ADC-12 aluminum matrix.

### 3.2. Microhardness Survey

An increase in microhardness of the developed surface composites was found with respect to increment in vol. % of  $\text{Cr}_3\text{C}_2$  dispersed onto the surface of ADC-12 aluminum alloy and the results are plotted in figure 7. This increment in hardness value of the surface composites fabricated through FSP route can be attributed to mainly three factors: (i) presence of hard ceramic particles dispersed onto the surface (ii) grain size reduction as a result of FSP and (iii) variations in heat transfer. Similar findings are reported by suganya et. al [21]. The properties of such matrix and reinforcement led to quench hardening, thereby increasing the surface characteristics of the developed surface composites. The quench hardened sample taken for hardness survey, shown in figure 7 revealed a greater increase in hardness

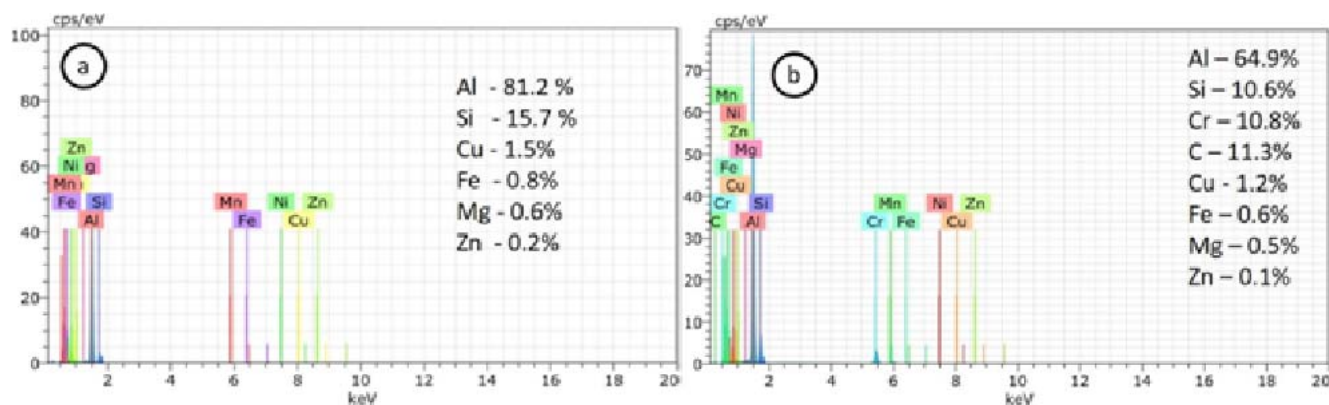


Fig. 6. EDS spectrum with norm.C(wt.%) (a) ADC-12 (b) ADC-12 Reinforced Cr<sub>3</sub>C<sub>2</sub>

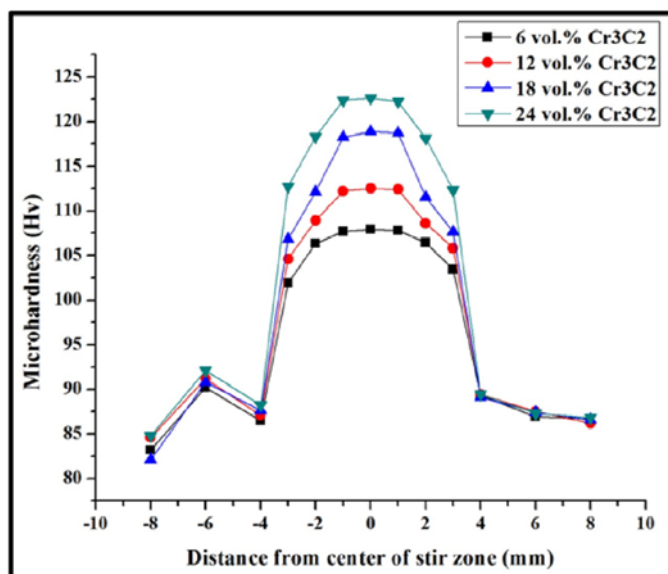


Fig. 7. Microhardness survey results

at the centre of stir zone. This is because of the large number of reinforcements of  $\text{Cr}_3\text{C}_2$  particles and the huge quantity of dislocations at particle matrix interface.

Dispersion of hard  $\text{Cr}_3\text{C}_2$  onto the surface of ADC-12 aluminum alloy enhances the hardness of the developed composite surface. Similar findings for various other carbides like TiC, SiC,  $\text{Al}_2\text{O}_3$  reinforced with matrix like copper and aluminium alloy respectively were reported by sabbaghian et al. [22], Barmouz et al. [23] and Sharifitabar [24]. This increase in hardness as an effect of particles dispersion can be attributed to the load carrying ability of the hard material. Again to this, the contradiction in thermal properties between the matrix metal and reinforced  $\text{Cr}_3\text{C}_2$  leads to the quench hardening effect in the thus developed surface composite enhancing the overall hardness in the processed stir zone. Reduction in the grain size of the developed AMC also affects the hardness [18] in a positive manner. With FSP, the grain size of the surface composites undergoes a tremendous decrement which in turn increases the hardness of the

metal. This phenomenon of hardness increment with grain size reduction is well established through Hall-Petch effect [19, 20]. This increment in hardness of the newly developed AMC may affect the other surface characteristics such as wear resistance in a positive manner.

#### 4. Conclusion

Friction stir processing was effectively used to disperse  $\text{Cr}_3\text{C}_2$  particles onto the surface of aluminum ADC-12 alloy and the microstructural and mechanical characterization of the same was carried out. Microstructural characterization of the developed AMC through optical micrographs exposed a refined grain size owing to the plastic deformation occurred during FSP routes. This dynamic recrystallization of matrix metal can enhance the mechanical characteristics of the developed surface composites. SEM micrographs of the developed AMC dispersed with varying vol. % of  $\text{Cr}_3\text{C}_2$  portrayed good bonding between the matrix and reinforcement. Again to this, the absence of cavities and detrimental phases at the matrix- reinforcement interface explains the strong bonding formed between the matrix and reinforcement. From the results, it is also evident that the  $\text{Cr}_3\text{C}_2$  particles have been dispersed uniformly onto the surface of ADC-12 matrix material. Microhardness survey analysis of the developed specimens depicted an enhanced hardness value and it was also accounted from the study that with an increase in vol. % of  $\text{Cr}_3\text{C}_2$ , the hardness of developed surface composites has been found to be increasing.

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